

as compared to about 7 percent for methyl methacrylate systems. The compressive strength is 36,200 pounds per square inch and tensile strength by diametral tests 6,000 pounds per square inch.

EXAMPLE 3

Dental filling compositions are prepared using pigmented siliceous materials as the inert inorganic filler. An example is a finely divided (preferably below 50 micron size) powdered borosilicate glass which is intimately mixed with about 0.1% of fluorescent pigment, about 0.01 percent of raw sienna and a few thousandths percent of very finely divided black and yellow pigments. This combination is found to be approximate normal tooth color in many cases and can be further colored when necessary. It is desirable to apply a treatment to this filler to promote wetting by resins. Silane treatment as with methacrylyl oxypropyl silane is appropriate.

One method for dispensing a composition is as a two part system of liquid and solid components which can be combined either from separate containers or are intermingled by rupture of a seal intervening between predetermined amounts. The dry component is formed from 69.65 parts of the above inorganic filler (including pigments) thoroughly mixed with 0.35 part of benzoyl peroxide. The resultant mixture is a dry powder.

The liquid composition is made from 29.50 parts of the bis methacrylate of 2,2-bis(p-hydroxyethoxyphenyl) propane:

$[\text{CH}_2=\text{C}(\text{CH}_3)\text{CO}-\text{OC}_2\text{H}_4\text{OC}_6\text{H}_4]_2\text{C}(\text{CH}_3)_2$ containing about 0.01 percent of methyl hydroquinone as inhibitor.

An adduct is made from phenyl salicylate (318 parts, 1.5 moles) with glycidyl methacrylate (113 parts, 0.83 moles) and about 4.3 parts of dimethyl-p-toluidine by heating at 60° C. for about 6 days followed by removal of unreacted salicylate first by Crystallization then by mild alkaline washes.

The bismethacrylate resin is thoroughly mixed with 0.29 part of the above phenyl salicylate adduct with glycidyl methacrylate and 0.21 part of N,N-bis-(2-hydroxyethyl)-3,5-xylidine. Aliquots in these weight proportions (70:30) suitably totalling about 0.5–2.0 grams, are placed in different parts of a package having a rupturable membrane. Mixing is effected by rupturing the membrane and kneading the solid and liquid together. This is done rapidly and the paste is used for filling previously prepared cavities in teeth. It sets rapidly to a sound filling.

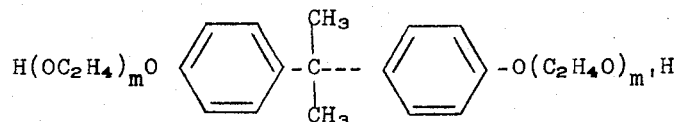
An alternative procedure is to prepare two pastes which are mixed mechanically or by spatulation in

equal amounts. The pastes may be prepared to have viscosities in the range of 100–8,000 centipoises and preferably about 1,000 to 4,000 centipoises.

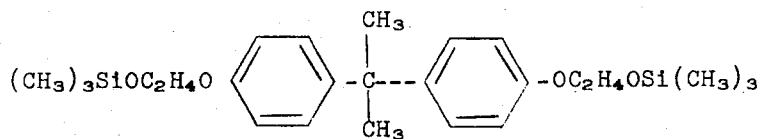
One paste is made containing 72 parts of the above siliceous filler with 27.27 parts of the above resin, 0.28 parts of the adduct of phenyl salicylate and glycidyl methacrylate and 0.45 parts of N,N-bis(hydroxyethyl)-p-toluidine. The second paste contains 25.58 parts of resin, 74 parts of siliceous filler 0.42 part of benzoyl peroxide and a trace (500 parts per million, 0.05 percent) of commercial butylated hydroxy toluene as an inhibitor. Equal portions of the two pastes are packaged, for example, in tubes and small amounts are dispensed and mixed as required. The resultant composition is effective for filling cavities.

EXAMPLE 4

A series of hydroxyethylated bisphenol A's is made of the general formula:



in which m plus m' ranges from about 1 to about 6. The procedure is that of Example 1, in which a 10 mole percent excess of ethylene oxide was used, but using various proportions of ethylene oxide, namely, 2, 2.6, 3.0, 4.0 and 6.0 moles per mole of bisphenol A. In order to determine the values of m and m' in the above formula small samples of each composition were converted to the silyl ethers, e.g.,



by reaction with N,O-bis-(trimethylsilyl)acetamide

$(\text{CH}_3)_3\text{Si}-\text{OC}(\text{CH}_3)=\text{NSi}(\text{CH}_3)_3$ followed by vapor phase or gas chromatography injecting at 260° C on to a 4 foot \times $\frac{1}{8}$ inches of 4% purified liquid methyl silicone on high density diatomite support (OV101 on chrome GS) column using helium at 25 ml./min. at 150°–300° C increasing 8° C per min. and flame ionization detection followed by isolation and mass spectrographic determination of molecular fragments. The amounts of product in percent by weight for the various values of $m + m'$ for the different mole ratios of ethylene oxide above are shown in the following table. Material not accounted for is a combination of loss and higher members of the series and is tabulated as "balance." The molecular weights and viscosities of the respective bis methacrylates also prepared as in Example 1 are included.

TABLE I

Moles C_2H_4 $m+m'$	2	2.6	3.0	4.0	6.0
1	8.7	2.8	0.5	—	—
2	79.6	54.2	35.5	11.6	8.0
3	5.6	27.2	37.6	27.3	20.6
4	0.9	6.7	16.2	30.2	26.9
5	—	—	4.2	15.4	22.9
6	—	—	—	6.0	13.4
Balance	4.8	2.6	6.1	7.0	8.2
Bis methacrylates molecular wt.	452	479	496	540	628
viscosity (cps)	1400	1050	940	690	600